IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re P	eatent Application of:)		
	RODNEY M. LAFOLLETTE, ET AL.)	Docket:	7310.C
Serial	No.:)	Art Unit:	
Filed:	14 August 2001)	Examiner:	
For:	MICROSCOPIC BATTERIES FOR MEMS SYSTEMS)	•	

TRANSMITTAL OF PARENT RELATED ART/

INFORMATION DISCLOSURE STATEMENT

Honorable Commissioner of Patents and Trademarks Washington, D.C. 20231

Sir:

Attached hereto for making of record in the U.S. Patent and Trademark Office in the aboveidentified application are the Related Art/Information Disclosure Statements submitted in parent U.S. Patent Application, as well as the PTO-948 forms.

Respectfully submitted,

Attorney for Applicants

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

PATENT APPLICATION SERIAL NO. 09/037 FILING DATE 10 March 1998
APPLICANT Rodney M. LaFollette et al.
GROUP ART UNIT 1741
EXAMINER Unknown
ATTORNEY'S DOCKET NO. 7310
TITLE "Microscopic Batteries for MEMS Systems"

SUPPLEMENTAL RELATED ART/INFORMATION DISCLOSURE STATEMENT

To: Commissioner of Patents and Trademarks Washington, D.C. 20231 From: L. Grant Foster

Foster & Foster L.C. 602 East 300 South

Salt Lake City, Utah 84102 Telephone: (801) 364-5633 Facsimile: (801) 355-8938

The Applicants do not believe that the related art set forth on the accompanying PTO-1449 forms is particularly germane and certainly such does not anticipate or make obvious the invention of the above-identified application.

To the best of the recollection of the Applicants and the undersigned, none is aware of any further patent or publication which might be deemed relevant to the claimed subject matter.

Furthermore, it should also be made of record that no exhaustive effort has been undertaken to locate, either on the part of the Applicants or the undersigned, any patent or publication which might in the past have been familiar to the Applicants or the undersigned and which may be contained in the files and/or among records concerning which none of the Applicants nor the undersigned has present recollection.

Restated, so far as the undersigned and the Applicants are able to presently recall, none is aware of any related art patents or publications believed to be germane in any way to the above-

identified application other than the patents mentioned below and said patents are only of general interest.

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Integrated circuits, including microelectronic circuits, have been used extensively, and have the advantage of small size and low production costs particularly when produced on a large scale. A class of integrated circuits that are of particular interest comprise microelectronic circuits having at least one MEMS device. MEMS may comprise complex engineering systems comprising microscopic mechanical elements, such as motors, pumps, relays, sensors, accelerometers and other components, which are powered by electrical energy. MEMS devices make possible controlled physical movement of tiny parts within miniature circuits.

MEMS devices have the potential to revolutionize computational technology. The concept of MEMS fabrication provides the promise of low cost comparable to the cost effectiveness in producing integrated electronic circuitry. MEMS can include sensing and actuating components. In defense systems, MEMS are expected to revolutionize the gathering, evaluation, and communication of militarily-significant information. "MEMS will create new military capabilities, make high-end functionality affordable to low-end military systems, and extend the operational performance and lifetimes of existing weapons platforms." (Department of Defense [DOD], 1995).

The great strength of MEMS as a technology fundamentally depends on: 1) the ability of MEMS to obtain increased functionality in a single, integrated system; 2) the low-cost, high-volume nature of MEMS fabrication; and 3) the overall reduction in size and mass of sensor/actuator systems. Heretofore, MEMS technology has typically focused on the need to fabricate MEMS and electronic devices that meet these three goals, but has failed to address the difficult problem of

electrical energy availability and management. The overall goals of many MEMS applications has not and will not be met unless one or more appropriate MEMS power sources are developed.

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While power and energy availability and management are problems for all integrated circuits, they are acute problems for MEMS. Many MEMS devices require periodic power pulses. Conventional wisdom has required and still requires that electrical power be supplied from relatively large, heavy external sources. Moving electrical current into an integrated circuit from such an external power source is difficult, and results in high power losses, particularly for a MEMS circuit where high capacities are required. Additionally, present MEMS devices must be continuously connected to the external power source. Thus, autonomous (self-contained), portable or remote operation of MEMS devices (such as MEMS sensors) is difficult if not impossible to achieve using an external fixed power source. It is reported that the 1994 market for MEMS was \$500M. The projections of potential future market, based on expected growth of existing markets and expansion into anticipated markets within a decade, are that the production of MEMS will approach \$3 billion per year.

A large fraction of MEMS production presently occurs in two areas, sensors and accelerometers (\$200M/year). These two markets are expanding very rapidly at present. In addition, other types of MEMS applications are rapidly emerging. Presently, none of these devices are using integrated batteries, because none exist, nor have they previously been invented and developed.

One analysis of the MEMS sensor and accelerometers applications are that nearly 100% of the sensor market, and approximately 30% of the accelerometer market, would use microscopic batteries, if they were available. On this basis, it is estimated that a market for microscopic batteries in these two fields would be \$50M/year, if such a product existed. Other significant markets would

also come into place if a microscopic battery could be provided, such that within a decade microscopic batteries, would have a market of over \$100M/year.

While the storage of energy in miniature, rechargeable devices for MEMS application is contrary to the state-of-the-art, a long term unsatisfied need for such has existed. If miniature energy sources were inventively created, significant advantages would be obtained, which are not presently available. First, more autonomous MEMS devices could be produced because the present dependency on continuous supply of power from an external source would be overcome. Second, significant improvement in energy efficiency would result. The supply of electrical energy would be at low power, stored temporarily, and then released at higher power levels in close proximity to the point of use, thus reducing overall power losses. Third, the cost of the MEMS system its integrated power supply would be lowered by reducing the complexity of electrical connections. Presently, it is difficult if not impossible to effectively store energy locally within a MEM system. Miniature capacitors have unacceptable useful discharge times (and hence unacceptable energy storage capacity). Fourth, cells can selectively be arrayed in series and in parallel to achieve different (and variable) combinations of operating voltage and capacity.

Further, unitary simultaneous formation, for example, of a microcircuit, one or more MEMS devices and a microscopic battery would provide a substantial advantage.

Presently. batteries for MEMS devices are unsatisfactory external power sources, which undesirably contributing to both the overall weight and volume of the MEMS device, and have other disadvantages. There are two primary reasons for this. The first primary reason is the size of the batteries. The smallest commercial batteries are the button-shaped energy cells used in watches, calculators and hearing aids. These are huge when compared with the MEMS to which such an

external battery heretofore has supplies power. The second primary reason is that the need for energy supply in MEMS is at a relatively high power level. High power is often needed to produce mechanical movement in MEMS devices. Commercially available batteries typically maximize the amount of energy they store, as opposed to providing high power release of stored energy. Consequently, conventional external batteries must be overly large in order to supply the power levels required by the circuit.

A further limitation of present commercially available external batteries for MEMS is that no small batteries are rechargeable batteries. Rechargeability is mandated by many MEMS applications.

Table 1 of this application compares the characteristics of several power source solutions with respect to size, weight, capacity, and assembly difficulty. Table 2 of this application is a partial list of potential DOD applications for MEMS taken from the text "Microelectromechanical Systems: A DOD Dual Use Technology Industrial Assessment" (DOD, 1995), together with an indication of the power source requirements for the majority of applications in the given area. As stated above, a significant portion of MEMS production presently occurs in two areas, sensors and accelerometers. Military applications for remote sensors and accelerometers include: safing and arming of fuses; friend or foe identification; embedded sensors for system integrity monitoring; communications systems monitoring, such as with satellites; low power mobile displays; flexible sensing surfaces; and numerous others.

Many of the application areas in Table 2 will require an integrated or integrable microscopic battery power. In general, systems that require mobile, autonomous, extensively-integrated sensors will require microscopic batteries. A requirement for mobility excludes standard wired power

sources. A requirement for autonomy excludes primary battery systems that cannot provide power to integrated systems for extended periods. Requirements for small size, extensive integration and large numbers of units exclude the use of coin-type or standard format batteries because of the difficulty of mounting such batteries into the format required by integrated systems. Microscopic batteries, once available, will have performance advantages that will prove to be critical to specific system applications, such as multiple, definable voltage levels, lower power requirements, and better power distribution.

Figure 17 of this application contrasts the power and energy capabilities of several prior energy storage technologies. Traditional electrostatic capacitors have the highest peak specific power (up to 10⁴ kW/kg) of prior technologies. The two major disadvantages of electrostatic capacitors are low specific energy and exponential discharge behavior. Specific energies are 0.01-0.1 J/g. The exponential decay in power output vs. time is not suitable for applications which require a flatter discharge profile. The advantage of electrostatic capacitors is that high voltages are possible, limited only by the ability of the dielectric material to sustain the voltage. The surface areas of the capacitor plates, where the charge is stored, are not high. Efforts to improve specific energy by increasing the plate areas have not achieved the objective.

Double layer capacitors, which have existed for decades (Becker, 1957) are not capable of high potential differences (<3 V, as opposed to > 10 V for electrostatic capacitors), but through the use of high surface area material in the electrodes, energy densities can be made to be much higher than electrostatic capacitors. High surface area carbon and sulfuric acid are the most common electrode material and electrolyte. Specific energies of 0.05-0.8 J/g are considered typical (Boos, 1970; Boos et al., 1971; Currie et al., 1985; Boos and Metcalf, 1972 and Isley 1972; Selover et al.,

1977; Rose, 1988; Rose, 1989). Peak specific powers are typically 10 - 30 W/g. Miniaturization of these very high surface area materials, however, would be difficult, if not impossible.

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A more thorough treatment of recent advances in capacitor technology are given by Raistrick (1992) and Oxley (1988). Progress has been made through the use of improved electrodes and electrolytes, but attempts to provide a combination of very high specific power (> 50 W/g) and specific energy (>30 J/g) have failed using capacitor technology.

A variety of electrochemical capacitors exist, which have been developed, and which have been substantially improved during the past decade. These range in their mode of operation from double-layer capacitors, which strictly use non-faradaic processes, to devices which are somewhat similar to batteries and which use faradaic reactions to release energy. Intermediate devices exist between pure double layer capacitors and batteries in their operation. During discharge of these devices, the double-layer releases a charge, but the electrode surfaces themselves also undergo faradic (charge transfer) reactions. Hence, these intermediate devices employ both faradaic (bulk) and non-faradaic (surface) reactions, which increase the energy which can be stored in the cell.

In contrast to capacitors, traditional secondary (rechargeable) batteries, which store energy in chemical form, have the highest specific energy of the technologies presented in Figure 5 (i.e. 90 - 400 J/g for existing Systems, up to 3000 J/g for some systems under development). Batteries have normally been designed to maximize the specific energy, at the expense of the specific power. Typical specific power values are low, i.e. 0.03 - 0.3 W/g (Linden, 1984).

Of the available electrical energy storage technologies, batteries are probably the leading candidate for use in MEMS. As shown in Figure 17 of this application, batteries can be designed to provide adequate levels of both power and energy. The major obstacle in using batteries in

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Internal batteries must be microscopic, not macro-scopic. Dimensions must be in micrometers,

rather than centimeters, and good specific power and specific energy must be available. Presently,

the smallest external batteries available commercially are of the order of 0.1 to 1 cm³ in volume and

1 to 3 g in weight. For example, button cells employing a variety of electrochemical couples (such

as silver/zinc, zinc/air, and lithium/manganese dioxide), are built which are approximately 1.06 cm²

in cross-sectional area, are 0.54 cm in height, and weigh 1 to 3 g (Linden, 1984). The open circuit

potential of these single cells is 1.5 to 3.0 V. The highest capacity batteries in this class can deliver

1440 C of energy, with a specific energy of 100 -1000 J/g (400 - 3200 J/cm³). The biggest difficulty

in their use to power MEMS, along with their size and weight, is the fact that all such batteries are

primary and are not secondary or rechargeable batteries.

Batteries for internal MEMS applications would need to have several important characteristics. First, many MEMS applications require the capability of large numbers of repeated charge/discharge cycles. Second, they must have a minimum of internal resistance to limit energy losses during battery operation. Third, they must be robust, so that changes in temperature, pressure, and other conditions do not damage performance. Fourth, MEMS batteries must be produced in large quantities, at low cost, and low rejection rate.

Recently, efforts have been made to provide smaller secondary (rechargeable) batteries which can operate at very high efficiency. Such smaller secondary batteries are far larger than microscopic circuits. Smaller bipolar lead acid batteries have been built and demonstrated, which had open circuit potentials of 2 - 8 V (1 - 4 V per individual cell). (LaFollette 1988; LaFollette and Bennion, 1990). These batteries were designed for high efficiency to produce very short bursts (0.1 - 100 ms)

of very high levels of power. These batteries produced up to 5A for 1 - 2 ms. for a power output of 35 W during that time. Their specific energy was approximately 70 J/g which, though modest by battery standards, is far better than capacitors. Peak specific power was 200-800 W/g (800 - 3000 W/cm³). Typical values for commercially available batteries are 0.1 W/g or 0.4 W/cm³). This specific power was achieved through the use of an efficient bipolar cell design, and the use of lightweight, high performance cell components. These batteries can also deliver multiple, high power discharges without a significant recharge (LaFollette, 1995).

While these efforts at building smaller batteries represent a decrease of two orders of magnitude in battery size from traditional batteries, the batteries in question are large when compared to the microscopic size needed to provide internal circuit power to a MEMS. What is required is an entirely new class of batteries (i.e. microscopic batteries) with peak specific powers much higher than present batteries, with specific energies many times that of capacitors, and which are built on a microscopic scale suitable for internal integration either into an existing MEMS, for retrofit purposes, or unitarily fabricated as part of the MEMS, for original manufacturing purposes.

The art includes certain thin-film batteries, which are also large by microelectronics standards. Included in this category are lithium batteries which may be able to provide high specific energy (Levasseur et al., 1989). The first totally thin-film rechargeable lithium battery was a Li/TiS₂ cell built by Kanehori, et al (1983). Since that time a variety of cells with different electrolyte and cathode materials have been made. In spite of their differences, all of these thin-film batteries use an evaporated layer of metallic lithium as the anode.

The most common electrolyte used in solid-state lithium batteries is a lithium glass. For example, xLi₂O-yB₂O₃ or xB₂O₃-yLi₂O- zLi₂SO₄ (Jones et al., 1994; Levasseur et al., 1989;

Balkariski et al., 1989) may be used. These glasses are typically sputter-deposited at a thickness less than 5μm. The resulting electrolyte layers have room temperature ionic conductivities ranging from 10° to 10° S/cm, depending on the composition of the electrolyte (Jones et al., 1994). In contrast, the ionic conductivity of a 5M KOH electrolyte is approximately 0.5 S/cm. One of the key problems with the lithium glass electrolytes has been an absence of long term stability in contact with metallic lithium (Bates, et al., 1993). This problem has been pursued by the Eveready Battery Company (EBC). A thin layer of LiI was deposited by vacuum evaporation between the lithium electrode and the glassy electrolyte (Jones et al., 1994). The LiI, however, has a conductivity which is less than 1/100th of the glassy electrolyte and was, therefore, kept as thin as possible. An alternate attempt to solve the stability problem was recently undertaken by Oakridge National Laboratories (ORNL), which developed a LiPON electrolyte which is stable when in contact with lithium (Bates, et al. 1993).

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A common characteristic of cathodes used in thin-film lithium batteries is that they are all lithium intercalation compounds which have open channels through which Li⁻ ions can diffuse without severely disrupting the surrounding framework (Bates, et al., 1993). Two cathode materials which have been used in recent thin-film cells assembled by ORNL and EBC are V₂O₅ and TiS₂, respectively. While other cells are also described in the literature, these two cells appear to be representative of the current state-of-the-art.

Oakridge National Laboratories has recently assembled batteries with TiS_2 , V_2O_5 , and Mn_2O_4 cathodes which incorporate a LiPON electrolyte developed at Oakridge (Bates, et al, 1993). Of these, the Li/ V_2O_5 cell exhibited the best performance with a capacity of 120 μ A/cm². As with all intercalation electrodes, the cell voltage decreased steadily during constant current discharge, owing

to insertion of lithium ions into the cathode. The cell was discharged from an open circuit voltage of 3.9 V to a cutoff voltage of 1.5 V at current densities of $100 \,\mu\text{A/cm}^2$. According to the authors, cathode utilization decreased by only a few tenths of a percent or less per cycle. The authors also concluded that the cathode was the major contributor to the battery resistance.

The battery made by EBC uses sputter-deposited TiS_2 as a cathode and has an open circuit voltage of 2.5 when the cell is fully charged. This cell is typically operated in the potential range from 2.5 to 1.8 V. The cell may vary from 8 to 12 μ m in thickness and have a capacity between 35 and 100 μ Ah/cm². This battery has a long cycle life. EBC batteries routinely complete more than 1000 cycles at current densities as high as 300 μ A/cm² and have actually cycled more than 10,000 times at a current density of 100 μ A/cm². These batteries are also able to deliver current pulses of two seconds duration at current densities of approximately 2 mA/cm². By comparison, four cell bipolar lead acid batteries have been constructed with a much larger total thickness of about 400 microns which discharged at a current density of 10 - 25 A/cm² for short duration pulses, over four orders of magnitude greater than the EBC cell. (LaFollette and Bennion. 1990)

Because lithium is highly reactive, it is essential that lithium batteries be sealed with a protective coating that is impermeable to both gas and water.

Lithium cells can also be assembled with a solid ion-conducting polymer as the electrolyte instead of the glass electrolytes (Owens, 1995). Such cells are not really thin-film cells since the thickness of the electrolyte (~50 - 100 µm) is typically much greater than that of a 1 µm thickness used in the thin-film cells. Polyethylene oxide was the initial polymer used in lithium polymer electrolyte cells. However, other polymeric electrolytes with increased room temperature conductivity have since been developed. The long term stability of the polymer and the formation

of resistive layers at the metallic lithium/polymer interface are both issues of concern with respect to these cells.

Thin-film batteries, other than those based on lithium, have also been investigated. In particular, silver and copper systems have been examined because of the high ionic conductivity of silver- and copper-based solid electrolytes (Julien & Nazri 1994; Levasseur, et al. 1989). By and large, these batteries have been found to be impractical owing to their relatively high cost and low energy density. One study-of interest was performed by Takahashi and Yamamoto who fabricated a Ag/A₃SI/I₂,C cell (Takahashi & Yamamoto, 1971). Six of these cells were stacked entirely by vacuum evaporation and provided an OCV of 1.2 V at 25°C. The cells were discharged at a high rate of 10 mA/cm². Unfortunately, the cells are not without problems as the iodine oxidizes the solid electrolyte. Reference may be made to Levesseur, et al (1989) and Julien & Nazri (1994) for information on other types of nonlithium thin-film batteries.

Fuel cells are limited in their power output and specific energy due to the need for manifolding of individual cells to introduce fuel and oxidant into the cells and remove reaction products. The complexity of these systems would seem to preclude their consideration for adaption to microscopic size. Dyer has reported a proton exchange membrane fuel cell which is only 0.2 cm² in area (Dyer. 1990). The fuel cell can be mounted (according to the Dyer disclosure) directly onto a printed circuit. It consisted of a thin-film platinum electrode (< 0.0001 cm) mounted on a substrate, a gas-permeable, proton exchange membrane separator which is only 0.0005 cm thick over this lower electrode, and a porous platinum electrode on the other side of the membrane. A mixture of hydrogen and oxygen is introduced into the vessel containing the fuel cell. Apparently, the membrane allowed hydrogen gas to move to the inner electrode, but restricted much of the oxygen

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mobility, thus allowing the fuel cell to operate. Power output was low (<0.005 W/cm²), probably due to the diffusion resistance of the hydrogen transport to the inner fuel cell. The energy efficiency was probably also quite low, due to recombination of hydrogen and oxygen at the outer electrode. Power output from this fuel cell was low. While this technology is certainly promising, they can't store or deliver adequate energy for use as a microscopic source of electrical power for MEMS and other microcircuits. Fuel cells, of course, are difficult to recharge.

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Previous efforts have been made to integrate a satisfactory source of electrical power and a MEMS. A number of different thin-film (but large area) batteries have been documented in the literature. Of these, rechargeable lithium batteries have shown the best performance, as demonstrated by excellent cycle life and shelf life. At least one notable effort to apply such batteries to use in conjunction with MEMS has been made by workers at Oak Ridge National Laboratory (Bates, et. al., 1993).

The lithium batteries are limited to low discharge rates. Therefore, the area of these thin-film batteries must be large in order to increase the power available from the battery to acceptable levels. Also, lithium is very reactive with water and O_2 so that such batteries must be completely isolated from the environment to be useful. It is believed that the lithium batteries are relatively expensive to build. For example, the EBC battery uses TiS_2 , which is a high cost cathode material.

It can be seen that if an integratable microscopic battery were made available it would fill an long existing, unsatisfied need in the MEMS and microelectronic technologies described above.

While the Applicants and the undersigned attorney believe PTO-1449 patents and publications are of general interest only, it is respectfully requested the Examiner make his or her own independent search and review to determine for himself or herself the extent to which the cited

publications and enclosed patents and any others located in the search are deemed to be relevant, if at all, to the presently claimed invention of the above-identified application.

Respectfully submitted.

Dated: 14 Myst 1998

L. Grant Foster Registration No. 33,236 Form PTO-1449

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTY, DOCKET NO., 7310

SERIAL NO.: 09/037,801

LIST OF ART CITED BY APPLICANT

APPLICANT Rodney M. LaFolleite, et al.

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APPLICANT, Rodney M. LaFollette, et al.

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U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

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	2	Stout, M.G., et al., 20 (1994)	. "A Multichip	p Module Implementation of a Neura	al Network," <u>Proceedi</u>	ngs of IEEE	Multic	chio M	odule Confere	ence, p.
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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

PATENT APPLICATION SERIAL NO.	09/037 901
FILING DATE	Moreh 1000
INVENTORS Rodney M. La	iviarch 1998
EXAMINED ROdney M. La	rollette et al.
EXAMINER	Unknown
ART UNIT	1741
ATTORNEY'S DOCKET NO.	7310
TITLE	AS Systems"

SECOND SUPPLEMENTAL RELATED ART/ INFORMATION DISCLOSURE STATEMENT

To: Commissioner of Patents

and Trademarks
Washington, D.C. 20231

From:

L. Grant Foster

Foster & Foster L.C. 602 East 300 South

Salt Lake City, Utah 84102 Telephone: (801) 364-5633 Facsimile: (801) 355-8938

The citations listed on attached PTO Form 1449 may be material to the examination of the subject application. Applicants submit copies of these references in compliance with the duty of disclosure defined in 37 C.F.R. §§1.56, 1.97-1.99. The Examiner is requested to review and make these references of record in the above-referenced application.

Pursuant to 37 C.F.R. §1.97(c), Applicant submit that no item of information contained in the Information Disclosure Statement was cited in a communication from a foreign patent office in a counterpart foreign application and, to the knowledge of the undersigned after making reasonable inquiry, neither was it known to any individual having a duty to disclose more than three months prior to the filing date of this information disclosure statement. Therefore, no fee is required for the Examiner to consider the references cited herein.

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Applicants consider the invention, as claimed in the application, to be patentable over the cited references. Applicants respectfully request expeditious examination and a favorable action on the merits.

Date: 20 MGV97 1998

Respectfully submitted.

.. Grant Foster Reg. No. 33,236 Form PTO-1449

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

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SERIAL, NO., 09/037-301

LIST OF ART CITED BY APPLICANT

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APPLICANTS Rodney M. Latorlette et al

FILING DATE 10 March 1998 GROUP: 1741 U.S. PATENT DOCUMENTS *Examiner Document Date Name Class Sunctass Filing Date Initial Number If Appropriate AAΑB AC ΑĐ AEΑF AGAHΑĹ AJΑK FOREIGN PATENT DOCUMENTS Document Date Country Class Subclass Translation Number Yes Nο AL AM ANAO AΡ OTHER RELATED ART (including Author, Title, Date, Pertinent Pages, Etc.) Boos, D.L., Adams, H.A., Hacha, T.H., Medicalf, J.El, "A 3 Cubic Inch 200,000 Microfarad Capacitor," Proceedings of the 21st Electronic Components Conference, p. 38 (1971). 2 NASA Tech Briefs, "Electronic Components and Circuits," March 1998, pp. 46-46 3 EXAMINER DATE CONSIDERED *EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609; Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re F	Patent Application of:)		
	RODNEY M. LAFOLLETTE ET AL.)	Docket:	7310
Serial	No.: 09/037,801)	Art Unit:	1741
Filed:	March 10, 1998)	Examiner:	Unknown
For:	MICROSCOPIC BATTERIES FOR MEMS SYSTEMS)		

SECOND SUPPLEMENTAL RELATED ART/INFORMATION DISCLOSURE STATEMENT

Honorable Commissioner of Patents and Trademarks Washington, D.C. 20231

Sir:

Enclosed is a copy of the Solid State Ionics article identified in the attached PTO-1449 form.

Respectfully submitted,

Lygin G. Foster

Attorney for Applicants

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I hereby certify that this correspondence is being deposited with the United States Postal Service as first class mail in an envelope addressed to: Commissioner of Patents and Trademarks, Washington, D.C. 2023 on October 15, 1998.

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Lynn G. Foster

Form PTO-1449

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

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SERIAL NO. 09/037,801

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APPLICANT Rodney M LaFollette et al.

GROUP 1741

FILING DATE, March 10, 1998

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	1	Solid State Ionics	. Volume 9 3ž 10 (Part II), 1993, North-Holland Publishing Comp	any - Amsterdam		·-	
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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re P	Patent Application of:)		
	RODNEY M. LAFOLLETTE, ET AL.)	Docket:	7310
Serial	No.: 09/037,801)		
Filed:	March 10, 1998)	Art Unit:	1741
For:	MICROSCOPIC BATTERIES FOR MEMS SYSTEMS)))	Examiner:	Unknown

RELATED ART/INFORMATION DISCLOSURE STATEMENT

Assistant Commissioner for Patents Washington, D.C. 20231

Sir:

The Applicants do not believe that the related art set forth on the accompanying PTO-1449 forms is particularly germane and certainly such does not anticipate or make obvious the invention of the above-identified application.

To the best of the recollection of the Applicants and the undersigned, none is aware of any further patent or publication which might be deemed relevant to the claimed subject matter.

Furthermore, it should also be made of record that no exhaustive effort has been undertaken to locate, either on the part of the Applicants or the undersigned, any patent or publication which might in the past have been familiar to the Applicants or the undersigned and which may be contained in the files and/or among records concerning which none of the Applicants nor the undersigned has present recollection.

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I hereby certify that this correspondence is being deposited with the United States Postal Service as first class mail in an envelope addressed to: Commissioner of Patents and Trademarks.

Washington, D.C. 20231, on July 17; 1998.

Restated, so far as the undersigned and the Applicants are able to presently recall, none is aware of any related art patents or publications believed to be germane in any way to the above-identified application other than the patents mentioned below and said patents are only of general interest.

Integrated circuits, including microelectronic circuits, have been used extensively, and have the advantage of small size and low production costs particularly when produced on a large scale. A class of integrated circuits that are of particular interest comprise microelectronic circuits having at least one MEMS device. MEMS may comprise complex engineering systems comprising microscopic mechanical elements, such as motors, pumps, relays, sensors, accelerometers and other components, which are powered by electrical energy. MEMS devices make possible controlled physical movement of tiny parts within miniature circuits.

MEMS devices have the potential to revolutionize computational technology. The concept of MEMS fabrication provides the promise of low cost comparable to the cost effectiveness in producing integrated electronic circuitry. MEMS can include sensing and actuating components. In defense systems, MEMS are expected to revolutionize the gathering, evaluation, and communication of militarily-significant information. "MEMS will create new military capabilities, make high-end functionality affordable to low-end military systems, and extend the operational performance and lifetimes of existing weapons platforms." (Department of Defense [DOD], 1995).

The great strength of MEMS as a technology fundamentally depends on: 1) the ability of MEMS to obtain increased functionality in a single, integrated system; 2) the low-cost, high-volume nature of MEMS fabrication; and 3) the overall reduction in size and mass of sensor/actuator

systems. Heretofore, MEMS technology has typically focused on the need to fabricate MEMS and electronic devices that meet these three goals, but has failed to address the difficult problem of electrical energy availability and management. The overall goals of many MEMS applications has not and will not be met unless one or more appropriate MEMS power sources are developed.

While power and energy availability and management are problems for all integrated circuits, they are acute problems for MEMS. Many MEMS devices require periodic power pulses. Conventional wisdom has required and still requires that electrical power be supplied from relatively large, heavy external sources. Moving electrical current into an integrated circuit from such an external power source is difficult, and results in high power losses, particularly for a MEMS circuit where high capacities are required. Additionally, present MEMS devices must be continuously connected to the external power source. Thus, autonomous (self-contained), portable or remote operation of MEMS devices (such as MEMS sensors) is difficult if not impossible to achieve using an external fixed power source. It is reported that the 1994 market for MEMS was \$500M. The projections of potential future market, based on expected growth of existing markets and expansion into anticipated markets within a decade, are that the production of MEMS will approach \$3 billion per year.

A large fraction of MEMS production presently occurs in two areas, sensors and accelerometers (\$200M/year). These two markets are expanding very rapidly at present. In addition, other types of MEMS applications are rapidly emerging. Presently, none of these devices are using integrated batteries, because none exist, nor have they previously been invented and developed.

One analysis of the MEMS sensor and accelerometers applications are that nearly 100% of the sensor market, and approximately 30% of the accelerometer market, would use microscopic

batteries, if they were available. On this basis, it is estimated that a market for microscopic batteries in these two fields would be \$50M/year, if such a product existed. Other significant markets would also come into place if a microscopic battery could be provided, such that within a decade microscopic batteries, would have a market of over \$100M/year.

While the storage of energy in miniature, rechargeable devices for MEMS application is contrary to the state-of-the-art, a long term unsatisfied need for such has existed. If miniature energy sources were inventively created, significant advantages would be obtained, which are not presently available. First, more autonomous MEMS devices could be produced because the present dependency on continuous supply of power from an external source would be overcome. Second, significant improvement in energy efficiency would result. The supply of electrical energy would be at low power, stored temporarily, and then released at higher power levels in close proximity to the point of use, thus reducing overall power losses. Third, the cost of the MEMS system its integrated power supply would be lowered by reducing the complexity of electrical connections. Presently, it is difficult if not impossible to effectively store energy locally within a MEM system. Miniature capacitors have unacceptable useful discharge times (and hence unacceptable energy storage capacity). Fourth, cells can selectively be arrayed in series and in parallel to achieve different (and variable) combinations of operating voltage and capacity.

Further, unitary simultaneous formation, for example, of a microcircuit, one or more MEMS devices and a microscopic battery would provide a substantial advantage.

Presently, batteries for MEMS devices are unsatisfactory external power sources, which undesirably contributing to both the overall weight and volume of the MEMS device, and have other disadvantages. There are two primary reasons for this. The first primary reason is the size of the

batteries. The smallest commercial batteries are the button-shaped energy cells used in watches, calculators and hearing aids. These are huge when compared with the MEMS to which such an external battery heretofore has supplies power. The second primary reason is that the need for energy supply in MEMS is at a relatively high power level. High power is often needed to produce mechanical movement in MEMS devices. Commercially available batteries typically maximize the amount of energy they store, as opposed to providing high power release of stored energy. Consequently, conventional external batteries must be overly large in order to supply the power levels required by the circuit.

A further limitation of present commercially available external batteries for MEMS is that no small batteries are rechargeable batteries. Rechargeability is mandated by many MEMS applications.

Table 1 of this application compares the characteristics of several power source solutions with respect to size, weight, capacity, and assembly difficulty. Table 2 of this application is a partial list of potential DOD applications for MEMS taken from the text "Microelectromechanical Systems: A DOD Dual Use Technology Industrial Assessment" (DOD, 1995), together with an indication of the power source requirements for the majority of applications in the given area. As stated above, a significant portion of MEMS production presently occurs in two areas, sensors and accelerometers. Military applications for remote sensors and accelerometers include: safing and arming of fuses; friend or foe identification; embedded sensors for system integrity monitoring; communications systems monitoring, such as with satellites; low power mobile displays; flexible sensing surfaces; and numerous others.

Many of the application areas in Table 2 will require an integrated or integrable microscopic battery power. In general, systems that require mobile, autonomous, extensively-integrated sensors will require microscopic batteries. A requirement for mobility excludes standard wired power sources. A requirement for autonomy excludes primary battery systems that cannot provide power to integrated systems for extended periods. Requirements for small size, extensive integration and large numbers of units exclude the use of coin-type or standard format batteries because of the difficulty of mounting such batteries into the format required by integrated systems. Microscopic batteries, once available, will have performance advantages that will prove to be critical to specific system applications, such as multiple, definable voltage levels, lower power requirements, and better power distribution.

Figure 17 of this application contrasts the power and energy capabilities of several prior energy storage technologies. Traditional electrostatic capacitors have the highest peak specific power (up to 10⁴ kW/kg) of prior technologies. The two major disadvantages of electrostatic capacitors are low specific energy and exponential discharge behavior. Specific energies are 0.01-0.1 J/g. The exponential decay in power output vs. time is not suitable for applications which require a flatter discharge profile. The advantage of electrostatic capacitors is that high voltages are possible, limited only by the ability of the dielectric material to sustain the voltage. The surface areas of the capacitor plates, where the charge is stored, are not high. Efforts to improve specific energy by increasing the plate areas have not achieved the objective.

Double layer capacitors, which have existed for decades (Becker, 1957) are not capable of high potential differences (<3 V, as opposed to > 10 V for electrostatic capacitors), but through the use of high surface area material in the electrodes, energy densities can be made to be much higher

than electrostatic capacitors. High surface area carbon and sulfuric acid are the most common electrode material and electrolyte. Specific energies of 0.05-0.8 J/g are considered typical (Boos, 1970; Boos et al., 1971; Currie et al., 1985; Boos and Metcalf, 1972 and Isley 1972; Selover et al., 1977; Rose, 1988; Rose, 1989). Peak specific powers are typically 10 - 30 W/g. Miniaturization of these very high surface area materials, however, would be difficult, if not impossible.

A more thorough treatment of recent advances in capacitor technology are given by Raistrick (1992) and Oxley (1988). Progress has been made through the use of improved electrodes and electrolytes, but attempts to provide a combination of very high specific power (> 50 W/g) and specific energy (>30 J/g) have failed using capacitor technology.

A variety of electrochemical capacitors exist, which have been developed, and which have been substantially improved during the past decade. These range in their mode of operation from double-layer capacitors, which strictly use non-faradaic processes, to devices which are somewhat similar to batteries and which use faradaic reactions to release energy. Intermediate devices exist between pure double layer capacitors and batteries in their operation. During discharge of these devices, the double-layer releases a charge, but the electrode surfaces themselves also undergo faradic (charge transfer) reactions. Hence, these intermediate devices employ both faradaic (būlk) and non-faradaic (surface) reactions, which increase the energy which can be stored in the cell.

In contrast to capacitors, traditional secondary (rechargeable) batteries, which store energy in chemical form, have the highest specific energy of the technologies presented in Figure 5 (i.e. 90 - 400 J/g for existing Systems, up to 3000 J/g for some systems under development). Batteries have normally been designed to maximize the specific energy, at the expense of the specific power. Typical specific power values are low, i.e. 0.03 - 0.3 W/g (Linden, 1984).

Of the available electrical energy storage technologies, batteries are probably the leading candidate for use in MEMS. As shown in Figure 17 of this application, batteries can be designed to provide adequate levels of both power and energy. The major obstacle in using batteries in MEMS is the size and weight of available batteries. To date, large external batteries have been used. Internal batteries must be microscopic, not macro-scopic. Dimensions must be in micrometers, rather than centimeters, and good specific power and specific energy must be available. Presently, the smallest external batteries available commercially are of the order of 0.1 to 1 cm³ in volume and 1 to 3 g in weight. For example, button cells employing a variety of electrochemical couples (such as silver/zinc, zinc/air, and lithium/manganese dioxide), are built which are approximately 1.06 cm² in cross-sectional area, are 0.54 cm in height, and weigh 1 to 3 g (Linden, 1984). The open circuit potential of these single cells is 1.5 to 3.0 V. The highest capacity batteries in this class can deliver 1440 C of energy, with a specific energy of 100 -1000 J/g (400 - 3200 J/cm³). The biggest difficulty in their use to power MEMS, along with their size and weight, is the fact that all such batteries are primary and are not secondary or rechargeable batteries.

Batteries for internal MEMS applications would need to have several important characteristics. First, many MEMS applications require the capability of large numbers of repeated charge/discharge cycles. Second, they must have a minimum of internal resistance to limit energy losses during battery operation. Third, they must be robust, so that changes in temperature, pressure, and other conditions do not damage performance. Fourth, MEMS batteries must be produced in large quantities, at low cost, and low rejection rate.

Recently, efforts have been made to provide smaller secondary (rechargeable) batteries which can operate at very high efficiency. Such smaller secondary batteries are far larger than microscopic

circuits. Smaller bipolar lead acid batteries have been built and demonstrated, which had open circuit potentials of 2 - 8 V (1 - 4 V per individual cell). (LaFollette 1988; LaFollette and Bennion, 1990). These batteries were designed for high efficiency to produce very short bursts (0.1 - 100 ms) of very high levels of power. These batteries produced up to 5A for 1 - 2 ms, for a power output of 35 W during that time. Their specific energy was approximately 70 J/g which, though modest by battery standards, is far better than capacitors. Peak specific power was 200-800 W/g (800 - 3000 W/cm³). Typical values for commercially available batteries are 0.1 W/g or 0.4 W/cm). This specific power was achieved through the use of an efficient bipolar cell design, and the use of lightweight, high performance cell components. These batteries can also deliver multiple, high power discharges without a significant recharge (LaFollette, 1995).

While these efforts at building smaller batteries represent a decrease of two orders of magnitude in battery size from traditional batteries, the batteries in question are large when compared to the microscopic size needed to provide internal circuit power to a MEMS. What is required is an entirely new class of batteries (i.e. microscopic batteries) with peak specific powers much higher than present batteries, with specific energies many times that of capacitors, and which are built on a microscopic scale suitable for internal integration either into an existing MEMS, for retrofit purposes, or unitarily fabricated as part of the MEMS, for original manufacturing purposes.

The art includes certain thin-film batteries, which are also large by microelectronics standards. Included in this category are lithium batteries which may be able to provide high specific energy (Levasseur et al., 1989). The first totally thin-film rechargeable lithium battery was a Li/TiS₂ cell built by Kanehori, et al (1983). Since that time a variety of cells with different electrolyte and

cathode materials have been made. In spite of their differences, all of these thin-film batteries use an evaporated layer of metallic lithium as the anode.

The most common electrolyte used in solid-state lithium batteries is a lithium glass. For example, xLi₂O-yB₂O₃ or xB₂O₃-yLi₂O- zLi₂SO₄ (Jones et al., 1994; Levasseur et al., 1989; Balkariski et al., 1989) may be used. These glasses are typically sputter-deposited at a thickness less than 5µm. The resulting electrolyte layers have room temperature ionic conductivities ranging from 10⁻⁹ to 10⁻⁵ S/cm, depending on the composition of the electrolyte (Jones et al., 1994). In contrast, the ionic conductivity of a 5M KOH electrolyte is approximately 0.5 S/cm. One of the key problems with the lithium glass electrolytes has been an absence of long term stability in contact with metallic lithium (Bates, et al., 1993). This problem has been pursued by the Eveready Battery Company (EBC). A thin layer of LiI was deposited by vacuum evaporation between the lithium electrode and the glassy electrolyte (Jones et al., 1994). The LiI, however, has a conductivity which is less than 1/100th of the glassy electrolyte and was, therefore, kept as thin as possible. An alternate attempt to solve the stability problem was recently undertaken by Oakridge National Laboratories (ORNL), which developed a LiPON electrolyte which is stable when in contact with lithium (Bates, et al. 1993).

A common characteristic of cathodes used in thin-film lithium batteries is that they are all lithium intercalation compounds which have open channels through which Li⁻ ions can diffuse without severely disrupting the surrounding framework (Bates, et al., 1993). Two cathode materials which have been used in recent thin-film cells assembled by ORNL and EBC are V₂O₅ and TiS₂, respectively. While other cells are also described in the literature, these two cells appear to be representative of the current state-of-the-art.

Oakridge National Laboratories has recently assembled batteries with TiS₂, V_2O_5 , and Mn_2O_4 cathodes which incorporate a LiPON electrolyte developed at Oakridge (Bates, et al, 1993). Of these, the Li/ V_2O_5 cell exhibited the best performance with a capacity of 120 μ A/cm². As with all intercalation electrodes, the cell voltage decreased steadily during constant current discharge, owing to insertion of lithium ions into the cathode. The cell was discharged from an open circuit voltage of 3.9 V to a cutoff voltage of 1.5 V at current densities of 100 μ A/cm². According to the authors, cathode utilization decreased by only a few tenths of a percent or less per cycle. The authors also concluded that the cathode was the major contributor to the battery resistance.

The battery made by EBC uses sputter-deposited TiS_2 as a cathode and has an open circuit voltage of 2.5 when the cell is fully charged. This cell is typically operated in the potential range from 2.5 to 1.8 V. The cell may vary from 8 to 12 μ m in thickness and have a capacity between 35 and 100 μ Ah/cm². This battery has a long cycle life. EBC batteries routinely complete more than 1000 cycles at current densities as high as 300 μ A/cm² and have actually cycled more than 10,000 times at a current density of 100 μ A/cm². These batteries are also able to deliver current pulses of two seconds duration at current densities of approximately 2 mA/cm². By comparison, four cell bipolar lead acid batteries have been constructed with a much larger total thickness of about 400 microns which discharged at a current density of 10 - 25 A/cm² for short duration pulses, over four orders of magnitude greater than the EBC cell. (LaFollette and Bennion, 1990)

Because lithium is highly reactive, it is essential that lithium batteries be sealed with a protective coating that is impermeable to both gas and water.

Lithium cells can also be assembled with a solid ion-conducting polymer as the electrolyte instead of the glass electrolytes (Owens, 1995). Such cells are not really thin-film cells since the



thickness of the electrolyte (~50 - 100 µm) is typically much greater than that of a 1 µm thickness used in the thin-film cells. Polyethylene oxide was the initial polymer used in lithium polymer electrolyte cells. However, other polymeric electrolytes with increased room temperature conductivity have since been developed. The long term stability of the polymer and the formation of resistive layers at the metallic lithium/polymer interface are both issues of concern with respect to these cells.

Thin-film batteries, other than those based on lithium, have also been investigated. In particular, silver and copper systems have been examined because of the high ionic conductivity of silver- and copper-based solid electrolytes (Julien & Nazri 1994; Levasseur, et al, 1989). By and large, these batteries have been found to be impractical owing to their relatively high cost and low energy density. One study of interest was performed by Takahashi and Yamamoto who fabricated a Ag/A₃SI/I₂,C cell (Takahashi & Yamamoto, 1971). Six of these cells were stacked entirely by vacuum evaporation and provided an OCV of 1.2 V at 25°C. The cells were discharged at a high rate of 10 mA/cm². Unfortunately, the cells are not without problems as the iodine oxidizes the solid electrolyte. Reference may be made to Levesseur, et al (1989) and Julien & Nazri (1994) for information on other types of nonlithium thin-film batteries.

Fuel cells are limited in their power output and specific energy due to the need for manifolding of individual cells to introduce fuel and oxidant into the cells and remove reaction products. The complexity of these systems would seem to preclude their consideration for adaption to microscopic size. Dyer has reported a proton exchange membrane fuel cell which is only 0.2 cm² in area (Dyer. 1990). The fuel cell can be mounted (according to the Dyer disclosure) directly onto a printed circuit. It consisted of a thin-film platinum electrode (< 0.0001 cm) mounted on a

substrate, a gas-permeable, proton exchange membrane separator which is only 0.0005 cm thick over this lower electrode, and a porous platinum electrode on the other side of the membrane. A mixture of hydrogen and oxygen is introduced into the vessel containing the fuel cell. Apparently, the membrane allowed hydrogen gas to move to the inner electrode, but restricted much of the oxygen mobility, thus allowing the fuel cell to operate. Power output was low (<0.005 W/cm²), probably due to the diffusion resistance of the hydrogen transport to the inner fuel cell. The energy efficiency was probably also quite low, due to recombination of hydrogen and oxygen at the outer electrode. Power output from this fuel cell was low. While this technology is certainly promising, they can't store or deliver adequate energy for use as a microscopic source of electrical power for MEMS and other microcircuits. Fuel cells, of course, are difficult to recharge.

Previous efforts have been made to integrate a satisfactory source of electrical power and a MEMS. A number of different thin-film (but large area) batteries have been documented in the literature. Of these, rechargeable lithium batteries have shown the best performance, as demonstrated by excellent cycle life and shelf life. At least one notable effort to apply such batteries to use in conjunction with MEMS has been made by workers at Oak Ridge National Laboratory (Bates, et. al., 1993).

The lithium batteries are limited to low discharge rates. Therefore, the area of these thin-film batteries must be large in order to increase the power available from the battery to acceptable levels. Also, lithium is very reactive with water and O₂ so that such batteries must be completely isolated from the environment to be useful. It is believed that the lithium batteries are relatively expensive to build. For example, the EBC battery uses TiS₂, which is a high cost cathode material.

It can be seen that if an integratable microscopic battery were made available it would fill an long existing, unsatisfied need in the MEMS and microelectronic technologies described above.

While the Applicants and the undersigned attorney believe PTO-1449 patents and publications are of general interest only, it is respectfully requested the Examiner make his or her own independent search and review to determine for himself or herself the extent to which the cited publications and enclosed patents and any others located in the search are deemed to be relevant, if at all, to the presently claimed invention of the above-identified application.

Copies of the PTO-1449 entries identified with an asterisks (*) are being acquired and will be sent upon receipt.

Respectfully submitted,

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APPLICANT: Rodney M. LaFollette, et al.

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